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PCT/E	P96/03531	August 9, 1996	September 29, 1995		
	OF INVENTION METHOD OF INVENTION METHOD OF INVENTION OF I	F CARRYING OUT CONTINUOURS ROTATING IN THE SAME			
	NT(S)FOR DOÆO/US HEIDEMEYER et al.				
		Designated/Elected Office (DO/EO/US) the fo	ollowing items and other information		
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Items I	1. to 16. below concern docume	nt(s) or information included:			
11 🕱	An Information Disclosure States	ment under 37 CFR 1.97 and 1.98.			
12. 🕱	An assignment document for reco	ording. A separate cover sheet in complian	ace with 37 CFR 3.28 and 3.31 is included.		
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VI FIGURE 2 MAR 1998 IN THE UNITED STATES PATENT & TRADEMAKE OFFICE 4

In Re Applic	eation of: Peter Heidemeyer	et al.)	
Case No.:	3210x198) Group Art Unit:	
Serial No.:)	
Filed:	herewith) Examiner:	
Title:	METHOD OF CARRYING OUT CONTINUOUS PREPARATION PROCESS ON TIGHTLY MESH EXTRUDERS ROTATING IN THE SAME SENSE)	
Assistant Co Washington,	emmissioner for Patents D.C. 20231		
Dear Sir:			
NEC follo they requi whic	HORIZATION TO PAY AND PETESSARY FEES. If any charges of wing Communication (including but may be paid out of our deposit a tree a Petition, please construe this at h is required to accompany the pay it icant hereby petitions for entry of the conth after the due date of, as	or fees must be paid in connection to not limited to the payment of account No. 12-0064. If this payment is muthorization to pay as the necessement. this Amendment which is mailed . Enclosed is check No	on with the issue fees), ayment also sary Petition within the, dated
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	EL, CONTE & SARET, LTD	John M. England, Jr. Scott B. Feder G. Peter Albert, Jr.	34813 33129 37268
Attorneys at Law 401 North Michig	gan Avenue	Lisa C. Childs Michael B. Allen William A. Meunier	39937 37582 41193

(312) 661-2100 c \jbc\rau\forms\198preli m

Preliminary Amendment

1. In the specification

On page 4, last line, after "illustration", insert -- and drawings--.

On page 5, line 5, insert

-- Fig. 3 is a vertical cut over the length of an extruder,

Fig. 4 is a horizontal cut over the length of the extruder, and

Fig. 5 is a cross-section through the extruder along the line V-V of Fig. 3.--

On page 5, line 11, after "200 to 400 rpm", insert

--Details of these commercial machines will be explained below, based on Figs. 3 to 5.--

On page 9, after line 5, insert the following paragraph.

--The twin-screw extruders used for the tests specified above will become apparent from the diagrammatic illustration of Figs. 3 to 5. The housing 1 of the extruder comprises several housing sections 2, 3, 4, which have flanges 5 by which they are detachably joined to each other by means of screws 6. The housing 1 comprises two cylindrical housing bores 7, 8 which are parallel to each other, intersect each other and have axes 9, 10 parallel to each other. Screw shafts 11, 12 are disposed in the housing bores 7, 8, each having a shaft 13, 14 which constitutes the core of the respective screw shaft 11 and 12, respectively, and a screw 15, 16 which is disposed on the core, spiralling in the form of a thread. On each screw shaft 11, 12, a screw root 17, 18 is defined by each screw 15, 16, by the shaft 13, 13 and the housing bore 7, 8. There is only a small gap between the screw 15, 16 and the respective housing bore 7, 8.

As seen in Fig. 4, the two screws 15, 16 rotate in the same direction. As seen in Figs. 4 and 5, the screws 15, 16 mesh, i.e. the screw 16 of the screw shaft 12 engages with the screw root 17 of the screw shaft 11 and vice versa.

An inlet hopper 20, through which the materials to be processed are supplied, is formed on the housing section 2 which is disposed downstream, related to a direction of conveying 19. Outlet tools, namely die heads 21 such as pelletizing dies, die plates and the like may be attached to the housing section 4 which is disposed downstream, seen in the direction of conveying 10.

The screw shafts 11, 12 are each driven in the same direction of rotation 25 by a motor 22 via a clutch 23 and a reduction and distribution gear 24. Each shaft 13 and 14, respectively, can be driven by a torque M_d . The distance \underline{a} of the axes 9, 10, the outer diameter D_a of each screw 15 and 16, respectively, and the screw inner diameter D_i are likewise seen in the drawing. D_a and D_i are identical for both screw shafts 11, 12.--

2. In the claims

Delete claims 2 to 5 without prejudice and insert the following claims 6 to 13.

6. A method of carrying out a continuous process of preparation of plastic material on an extruder, comprising

a housing,

at least two cylindrical housing bores, each housing bore having an axis, said axes being disposed parallel to each other, said housing bores intersecting each other and having a first end and a second end,

an inlet hopper being provided in the vicinity of said housing bores and opening into said housing bores,

a die head being disposed at said second end of said housing bores,

at least a first screw shaft and a second screw shaft being disposed in said at least first and second housing bores,

said at least first and second screw shaft being provided with a first screw and a second screw, respectively, each screw defining a screw root, said first screw and said second screw root tightly intermeshing and said second screw and said first screw root tightly intermeshing,

said first and said second screw each having a screw outer diameter D_a and a screw inner diameter D_i, said screw diameters being identical,

said at least first and second screw shaft each having a volumetric ratio of at least 1.5, wherein the volumetric ratio is defined by the screw outer diameter D_a divided by the screw inner diameter D_i , and

said first and said second axis having a distance a of axes,

a drive connectable to said at least first and second screw shaft to drive said at least first and second screw shaft in a same direction of rotation at a screw speed of rotation n_S and at a shaft torque M_d ,

wherein the method comprises the following steps:

feeding plastic material to be processed through the inlet hopper into said housing bores,

conveying said plastic material through said screw roots from said inlet hopper to said outlet,

driving each of said at least first and said second screw shaft at a screw speed of rotation n_s of at least 600 rpm, at a torque density $M_{d/a}$, of at least 11 Nm/cm³, wherein the torque density is defined by the shaft torque Ma [Nm] at each first and second shaft divided by the third power of the distance \underline{a} of the first and second axes of the first and second screw shaft.

- 7. A method according to claim 6, wherein said at least first and second screw shaft is driven at a screw speed of rotation n_S of at least 800 rpm.
- 8. A method according to claim 6, wherein the at least first and second screw shaft is driven at a screw speed of rotation $n_{\rm S}$ of up to 3000 rpm.

- 9. A method according to claim 6, wherein said at least first and second screw shaft is driven at a torque density M_d/a^3 of up to 15 Nm/cm³.
- 10. A method according to claim 6, wherein the plastic material is conveyed through said housing bores from said inlet hopper to said outlet at a mean product dwell time of less than 10 seconds.
- 11. A method according to claim 10, wherein the plastic material is conveyed through said housing bores from said inlet hopper to said outlet at a mean product dwell time of less than 2 seconds.
- 12. A method according to claim 6, wherein in addition to plastic material, pigments are fed into said housing bores.
- 13. A method according to claim 6, wherein said method is exercised on an extruder having a volumetric ratio of at least 1.55.

3. In the drawings

Please accept the proposed drawing change, which is the addition of Figures 3, 4 and 5, submitted under separate cover to the official draftsperson.

Applicant respectfully requests approval of the proposed drawing change.

Respectfully submitted,

LAFF, WHITESEL, CONTE & SARET, LTD.

Date: New 77, 1998

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09/043954

Method of Carrying out Continuous Preparation Processes on Tightly

Meshing Extruders Rotating in the same Sense

Specification

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The invention relates to a method of carrying out continuous preparation processes on tightly meshing extruders rotating in the same sense such as twin-screw and multi-shaft screw-type extruders.

Tightly meshing twin-screw and multi-shaft screw-type extruders rotating in the same direction are used for continuous kneading processes with or without melting. Frequently, continuous degassing, mixing and expanding processes are integrated, too, and there are cases when the extruders are also used for reactions.

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The products to be processed comprise plastics, resins, liquids, viscoplastic fluids, pulverulent and fibrous additives as well as food compounds. Discharging may take place for instance via filters and molding processes such as pelletization and section extruding.

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Extruders of the type mentioned at the outset have been known, in which the screw diameter amounts to as much as 340 mm. The throughputs range from 5,000 to 35,000 kg/h at a ratio between the screw outer diameter and screw inner diameter (D_a/D_i) of 1.18 to 1.25 and 1.4 to 1.6, respectively.

The ratio between torque and center distance³ of axes (m_d/a^3) - the so-called "torque density" - is in the range of 5 to 10. Depending on the size of the extruders, speeds of rotation are run, ranging from 200 to 500 rpm - in exceptional cases even up to 600 rpm.

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Customarily, the design of the extruders is based on the principle of geometric and torque related similarity. Geometric similarity exists when the ratio D_a/D_i is constant; torque related similarity exists when the ratio M_d/a^3 is constant.

In addition to melt temperature and dwell time, the shear rate within the screw root filled with melt is another decisive factor for the dispersing, mixing and homogenization grade of the processed product.

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In many processes the mixing, dispersing and homogenization grade will be the higher, the higher the shear rate. In today's state of extruder engineering, mean shear rates in the melting range of between 20 to 150 1/sec and mean product dwell times in the entire screw range of between 15 to 60 sec are customary in standard preparation processes.

In conventional extruders, the mean shear rates are restricted upwards by the screw speed of rotation and the so-called "volumetric ratio" represented by the ratio D_a/D_i . However, increasing shear rates are accompanied with higher specific values of the energy supplied, which may lead to inacceptably high melt temperatures. Together with high average dwell times of the product in the extruder, this may lead to quality reducing deterioration of the product in terms of thermal decomposition and cross-linking.

25 It is the object of the invention to put into practice quality increasing mean shear rates in the range of up to ≥ 1,000 1/sec accompanied with simultaneous reduction of the duration of action of temperature peaks in the product without possible occurrence of the difficulties described above.

K i f

This object is attained by the extruder being operated at a screw speed of rotation of at least 800 rpm while the so-called "torque density" (M_d/a^3) which can be induced is simultaneously increased by at least 11 Nm/cm³ and the volumetric ratio (D_a/D_i) is at least 1.5.

With the increased torque density (M_d/a^3) selected according to the invention of at least 11 Nm/cm³, the extruder can easily be operated at the high screw speeds of rotation without any inadmissibly high specific energy input resulting. Another advantage resides in a very high product throughput per time unit.

Suitably, the dwell time of the product in the extruder is less than 10 seconds.

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In keeping with another embodiment of the invention, the extruder is operated at a screw speed of rotation of up to 3,000 rpm, while the so-called "torque density" (M_d/a^3) that can be induced is increased by up to 15 Nm/cm³ and the volume ratio is greater than or equal to 1.55 and the mean product dwell time is less than 2 seconds. This will give especially short (mean) dwell times of the product in the extruder as a result of the high throughputs then possible.

The short product dwell times of 1 to 10 sec resulting from the high screw speeds of rotation and the high product throughputs simultaneously reduce the tendency toward thermal decomposition or cross-linking of the products.

Within certain limits, an increase of the screw speed of rotation is also possible without an increase of the torque density (M_d/a^3) . However, the maximum screw speed of rotation is limited by the maximum upper limit of specific energy input which exists in every process and corresponds to the maximally permissible melt temperature (without deterioration of the product occurring).

The embodiment according to the invention of the method of the type mentioned at the outset helps open further fields of use.

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For instance, the method according to the invention can be used for the continuous masterbatching in the solids conveying range and for the grinding into powder of coarse-grained bulk material. Also, a combination of the two processes mentioned above is conceivable, i.e. a homogenization process of solids, which requires clearly less energy as compared with homogenization in the plastic phase.

Using the method according to the invention in reaction machines will moreover ensure efficient masterbatching of monomers and catalyst prior to the reaction in the incubation period.

Another advantage of the method according to the invention resides in that for instance pigments can be dispersed distinctly better during master-batching.

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In the following, the invention will be explained, based on diagrammatic illustration, in which

Fig. 1 is a representation of the "mean specific energy input",

Fig. 2 is a representation of the "material throughput and mean dwell time of the product in the extruder".

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Tests based on the method according to the invention were made on commercial two-shaft screw-type machines (two-shaft screw kneaders having tightly meshing screw shafts rotating in the same direction), the structure of the machine (screw geometry, mixing and kneading elements) being left as used so far for the respective plastics preparation process at customary speeds of rotation of 200 to 400 rpm.

In the tests, use was made of screw speeds of rotation by far exceeding 1000 rpm, and surprisingly it was found that upon simultaneous increase of the induced torque density to 11 to 14 Nm/cm³, no substantial increase of material temperature occurred. Even with an increase of the material temperature (for instance $PC > 350^{\circ}C$) to some unusually high melt temperature, there is no deterioration of the product, since the dwell times in the extruder are by far less than 10 s due to the method according to the invention.

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Fig. 1 diagrammatically illustrates the context between the screw speed of rotation (shear rate) and the specific energy supplied for varying torque densities Md/a³. Under the condition that the available torque is utilized, higher throughputs result while torque density increases (at a constant speed of rotation). Noticeably, reduced energy inputs and thus lower melt temperatures result from an increased torque density. On the other hand, an increase of the screw speed of rotation will generally lead also to increased

material throughput, however, at a given torque density, this throughput is connected with an increased energy input.

Fig. 2 illustrates the dependency of throughput and dwell time. It shows that while the throughput increases, the time during which the material is exposed to high temperatures is clearly reduced.

Tests carried out have shown that even a material temperature which, from experience so far, simply had to lead to reduced quality in the product will not mean quality impairment if the time of exposure is sufficiently short. However, sufficiently short dwell times can only be obtained by increased throughputs which, again, can only be realized by an increase of the possible torque, since otherwise, at a given (high) speed of rotation, the driving power of the machine will no longer be sufficient.

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As can also be seen in Fig. 1, an increase of the speed of rotation within certain limits is possible even without an increase of the torque density. The maximum upper limit, adherent to every method, of specific energy input (e_{specmax} corresponds to the maximally permissible melt temperature without deterioration of the product at a given dwell time) restricts this speed of rotation.

As a rule, today's machines exhibit D_a/D_i values ranging from 1.4 to 1.6 and Md/a³ values between 5 and 10. Depending on the layout, the operating speeds of rotation range from 200 to 500 rpm, even to 600 rpm in exceptional cases.

The throughput and the quality of the compounded product depend on the screw geometry utilized, on the speed of rotation and the maximum torque of the machine.

5 It is the object of every compounding job to obtain a homogenous final product - as a rule by incorporation of additives. The additives and any existing inhomogeneities must be dispersed in the machine and incorporated distributively. Dispersing particles requires more or less elevated shear stresses, which have to be imparted to the particles by way of the surrounding matrix. According to the equation

$$\tau = \eta * \overline{\dot{Y}} \tag{1}$$

- the shear stress τ results from the viscosity η of the matrix medium and the shear rate \dot{Y} there forced on. Therefore, in addition to the melt temperature and the dwell time, the shear rate \dot{Y} [1/sec] is a decisive factor for the dispersing, mixing and homogenization grade of the processed product in the screw root filled with melt.
- In a simplified consideration of the shear rate as a mean value from the quotient circumferential screw speed/spiral depth, the following applies (provided a 100 % degree of filling in the screw root):

$$\overline{\dot{Y}} = \frac{V_u}{\overline{h}} = \frac{D_a \cdot \Pi \cdot n_s}{(D_a - D_i) / 2} \tag{2}$$

or

$$\overline{\dot{\gamma}} = 2\pi * n_s * \frac{(D_a / D_i)}{(D_a / D_i) - 1}$$

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The following is true for many processes:

The higher the shear rate, the higher the mixing, dispersing and homogenization grade. With today's state of extruder engineering, mean shear rates in the melt range of 20 1/s to 150 1/s and mean dwell times of the product in the entire screw range of 15 to 60 s are customary in standard preparation processes.

In conventional extruders, the mean shear rates are restricted upwards by the screw speed of rotation and the "volumetric ratio" designated by D_a/D_i as seen from the equation (2).

However, according to the equation

$$\overline{e}_{spec} = \frac{1}{\rho_s} * \overline{\eta}_{(\dot{Y})} * \overline{\dot{Y}}^2 * \overline{t}$$
 (3)

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and

$$\overline{e}_{spec} = \frac{1}{\rho_s} \cdot \overline{\eta}_{(Y)} \cdot \overline{t} \cdot 4\pi^2 \cdot n_s \cdot \left[\frac{(D_a/D_i)}{(D_a/D_i) - 1} \right]^2$$

higher values of specific energy input e_{spec} will result in the case of increasing shear rates, which again may lead to inacceptably high melt temperatures, since the temperature increase of the melt is computed from the equation ΔT = e_{spec}/C_P (C_P = specific heat capacity). Combined with high average dwell times of the product in the extruder, a high shear rate may also lead to quality reducing deterioration of the product (thermal decomposition or cross-linking).

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In twin-screw extruders rotating in the same sense and having screw speeds of rotation in the range of 600 to 3000 rpm accompanied with the increase of the induced torque density to 11 to 15 Nm/cm³, quality increasing mean shear rates of up to 1000 1/s and simultaneous reduction of the duration of action of temperature peaks in the product can be realized by the method according to the invention.

Symbols Used:

	\overline{e}_{spec}	mean specific energy supplied [kWh/kg]
5	ī	mean dwell time of the product in the extruder [s]
	ρ	melt density [kg/m³]
10	$\overline{\dot{Y}}$	mean shear rate [1/sec]
10	$\bar{\eta}$	mean dynamic viscosity [Pa sec]
	D_a	screw outer diameter [mm]
15	\mathbf{D}_{i}	screw inner diameter [mm]
	$\overline{\mathbf{h}}$	spiral depth, mean value
20	n_s	screw speed of rotation [min ⁻¹] ([s ⁻¹])
20	M_{d}	shaft torque, related to 1 shaft [Nm]
	a	center distance of axes of the screw shafts [cm]
25	V_{u}	circumferential speed of the screw shafts [m/s]
	M_{d/a^3}	torque density, related to 1 shaft [Nm/cm³]
20	$\overline{\tau}$	shear stress [Nm/mm ²]
30	c_p	specific enthalpy [kJ/kg*K]
	m	material throughput [kg/h]
35	ΔΤ	material temperature increase [K]

 $|\mathbf{f}| = \mathbf{f}_{1,1} = \mathbf{f}$

Patent Claims

1. A method of carrying out continuous preparation processes on tightly meshing extruders rotating in the same sense such as twin-screw and multishaft screw-type extruders, **characterized in that** the extruder is operated at a screw speed of rotation of at least 800 rpm accompanied with a simultaneous increase of the so-called "torque density" (Md/a³) which can be induced of at least 11 Nm/cm³ and a volumetric ratio (Da/Di) of at least 1.5.

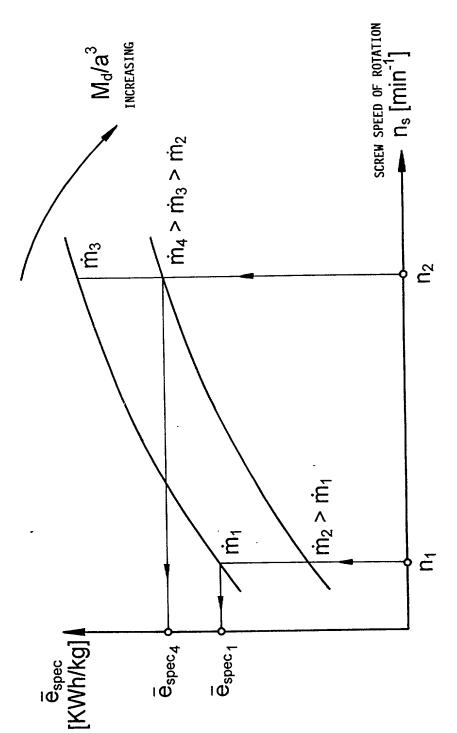
10

- 2. A method according to claim 1, characterized in that the mean product dwell time is less than 10 seconds.
- 3. A method according to claim 1, **characterized in that** the extruder is operated at a screw speed of rotation of up to 3000 rpm accompanied with a simultaneous increase of the so-called "torque density" md/a³ which can be induced of up to 15 Nm/cm³ and a volumetric ratio (Da/Di) equal to or greater than 1.55 and a mean product dwell time of less than 2 seconds.
- 4. Use of the method according to one or several of claims 1 to 3, characterized in that the method is used for the continuous mixing in the solid conveying range and/or for the grinding into powder of coarse-grained bulk goods.
- 25 5. Use of the method according to claim 4, characterized in that pigments are incorporated during masterbatching.

Abstract

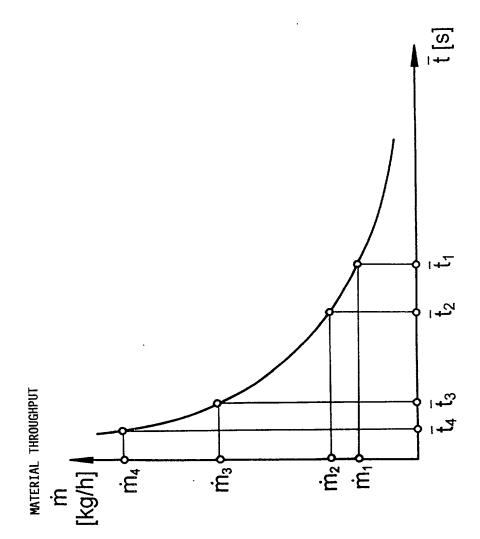
In a method of carrying out continuous preparation processes on tightly meshing extruders rotating in the same sense such as twin-screw and multi-shaft screw-type extruders, the extruder is operated at a screw speed of rotation of at least 800 rpm, accompanied with a simultaneous increase of the so-called "torque density" (M_d/a³) of at least 11 Nm/cm³ and a volumetric ratio (Da/Di) of at least 1.5.

1/2

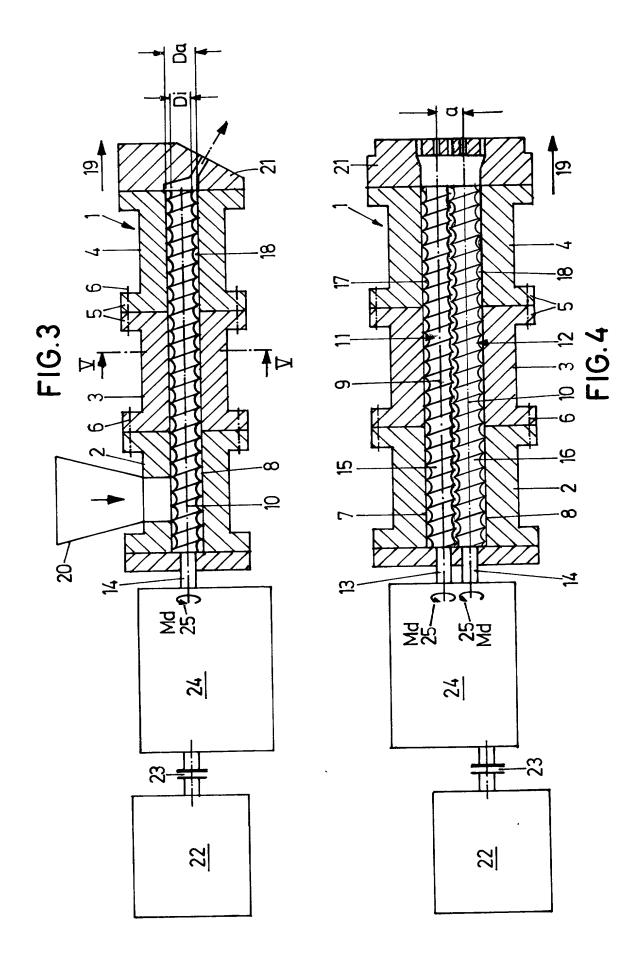


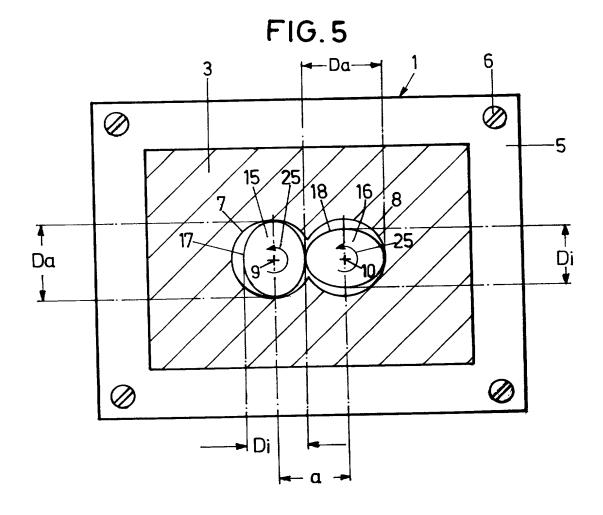
AVERAGE SPECIFIC ENERGY SUPPLIED

Fig. 1



AVERAGE DWELL TIME OF THE PRODUCT IN THE EXTRUDER





Declaration and Power of Attorney For Patent Application **English Language Declaration**

As a below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled Method of Carrying out Continuous

Preparation Processes on Tightly Meshing Extruders Rotating in the same Sense the specification of which (check one) X is attached hereto. was filed on. Application Serial No. $_$ and was amended on ___ (if applicable) I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above. I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a). I hereby claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed: Prior Foreign Application(s) **Priority Claimed** 195 36 289.6 Germany September 29, 1995 (Number) (Day/Month/Year Filed) (Country) (Number) (Country) (Day/Month/Year Filed) (Number) (Country) (Day/Month/Year Filed)

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

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	PCT/EP96/03531	August 9, 1996	
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	statements made on information ar were made with the knowledge th by fine or imprisonment, or both, u	nd belief are believed to be tru at willful false statements ar ander Section 1001 of Title 18	rn knowledge are true and that all ue; and further that these statements and the like so made are punishable of the United States Code and that he application or any patent issued
f	prosecute this application and transact Charles A. Laff (19787); J. Warren W Martin L. Stern (28911); Betrard LAS	ct all business in the Patent and Vhitesel (1 <u>6830)</u> ; Robert F. I. C (설계(전)(22128); Louis Altman (1	ollowing attorney(s) and/or agents(s) to Trademark Office connected therewith. Conte (20354): Larry L. Saret (27674); 9373); Barry W. Sufrin (27398): 林林 本. 3323科/and Jack R. Halvorsen (18394).
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end Correspondence to: LAFF, WHI linois 60611.	TESEL, CONTE & SARET,	401 North Michigan Avenue, Chicag

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In Re Applica	tion of: Peter Heidemeyer et al.)		
Case No.:	3210x198) Oroup Art Unit:		
Serial No.:)		
Filed:	herewith) Examiner:		
Title:	METHOD OF CARRYING OUT CONTINUOUS PREPARATION PROCESS ON TIGHTLY MESHING EXTRUDERS ROTATING IN THE SAME SENSE) ATTN: OFFICIAL) DRAFTSPERSON))		
Assistant Com Washington, I	CIAL DRAFTSPERSON missioner for Patents D.C. 20231			
Dear Sir:				
 [x] AUTHORIZATION TO PAY AND PETITION FOR THE ACCEPTANCE OF ANY NECESSARY FEES. If any charges or fees must be paid in connection with the following Communication (including but not limited to the payment of issue fees), they may be paid out of our deposit account No. 12-0064. If this payment also requires a Petition, please construe this authorization to pay as the necessary Petition which is required to accompany the payment. [] Applicant hereby petitions for entry of this Amendment which is mailed within themonth after the due date of, as payment of the necessary Petition fee. 				

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	Louis Altman	19373
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